A Small-displacement Monochromator for Microdiffraction Experiments

A monochromator has been designed and operated to perform microdiffraction experiments requiring very small displacements. This incident beam monochromator is capable of phase, texture, and strain measurements with micron or submicron spatial resolution in the 14 to 22 keV energy range on fine-grained polycrystalline materials.

The recent availability of ultra-brilliant third-generation synchrotron sources has created an opportunity to map the crystallographic phase, texture, and strain of materials with micron or submicron spatial resolution [1]. However, for fine-grained polycrystalline samples, new methods are required.

Laue diffraction offers a rapid method for determining crystal orientation without sample rotations. Laue diffraction can be extended to the measurement of deviatoric strain and plastic deformation with high-precision angular measurements. It can determine hydrostatic strain if the energy of one or more reflections is measured [1].

Various techniques have been used to measure the x-ray energies of microdiffraction Laue spots [2]. Compared with alternative techniques, an incident beam monochromator has both major technical advantages and major technical challenges. The chief advantage of an incident beam monochromator is intrinsically good energy resolution. For precision absolute-strain measurements, a monochromator with ~4 eV energy resolution and *even better absolute accuracy* at 20 keV is desirable.

Incident beam x-ray monochromators on third-generation synchrotron sources are challenged by the need for thermal and mechanical stability under a high-thermal-flux x-ray beam [3]. For x-ray microdiffraction it is important that any upstream optics maintain a constant offset as the beam is tuned or cycled between polychromatic and monochromatic conditions. For example, with focusing optics of magnification M, a beam displacement δ before the focusing optics results in an image motion of δM.

Fortunately, the very nature of microdiffraction experiments simplifies the beam stability and thermal load problems; typically only about 0.1% of the thermal load from a high-brilliance third-generation undulator is within the usable emittance for microdiffraction. As described in Ref. 3, this brings the total power on the first crystal of a monochromator into a range that can be handled by water-cooled optics. A detailed theoretical discussion of the anticipated thermal distortions on a water-cooled microbeam monochromator crystal is given in Ref. 3. In general, the distortions do not substantially degrade source brilliance.

To achieve the high precision required for microdiffraction, a rugged and mechanically stable design was adopted. This design has three key elements: (1) a thermally stable water-cooled incident beam vertical slit; (2) a rigid, two-crystal, small-displacement monochromator; and (3) a high-precision beam-defining vertical exit slit.

Particularly critical is the monochromator drive mechanism. This mechanism must provide an absolute energy reference. The two-crystal x-ray monochromator is based on flex pivot rotations. A rectangular cage is used to hold the first and second crystal subassemblies. The assembly is designed to allow for a chi (roll) tilt of the first crystal and for a $\Delta\theta$ (pitch) tilt of the second crystal. The overall cage

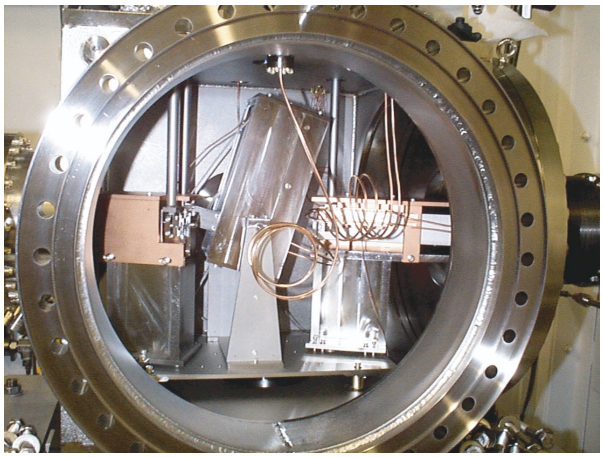

FIG. 1. View of the MHATT-CAT microdiffraction monochromator as seen through the main access flange. The tilted rectangular box in the middle of the monochromator holds the two Si (111) crystals.

assembly is driven by a Nanomover™ actuator, which is coupled to the crystal cage through a linear ball-bearing stage and two flex pivots.

The entire chamber is supported on parallel linear bearings with a stepping-motor-driven lead screw to laterally translate the monochromator crystals into or out of the incident beam. The assembled microbeam monochromator, with the main access flange removed, is shown in Fig. 1.

Energy calibration. The monochromator absolute energy scale and resolution were calibrated by measuring the near-edge structure of standard metal foil samples. Near-edge absorption edges of Cu, Mo, Sr, and Rh were measured and compared to the literature edge values. The a*bsolute calibration* of the monochromator was found to be better than ±0.5 eV over the 8 to 22 keV nominal range of the monochromator.

Hysteresis. Monochromator reproducibility and hysteresis were checked both over short (approximately ±1 keV) scans and over the entire range of the monochromator. As shown in Fig. 2, scans of less than 1 keV show no measurable hysteresis. Scans of ~10 keV show ~2 eV backlash. Note that the steps in the Mo edge scan are due to roundoff errors in the control software and do not appear below ~15 keV.

Warm-up. Because of the small thermal mass of the monochromator crystals and because of the small power load (about 1-4 W), there was no observable warm-up time for the instrument after beam turn-on or after the monochromator was inserted into the beam. Warm-up was checked by measuring the Mo edge immediately after insertion of the monochromator and 1 hr after insertion of the monochromator. No observable displacement of the edge position was detected.

Beam displacement. The beam displacement was checked by measuring the position of a standard gold wire with monochromatic and with polychromatic beams. Both the vertical and horizontal focal spot positions with the two beams were within the focal spot resolution.

Energy resolution. The energy resolution of the monochromator was inferred from the Cu near-edge spectra. The shape of near-edge features indicates an upper bound to the energy resolution of ~2 eV or better at 9 keV. This is near the theoretical Darwin width limited resolution of the Si (111) monochromator crystals.

In Table I, the design goals for a high-performance microbeam monochromator are compared to the measured performance. As can be seen, the monochromator meets or exceeds most goals. The one goal that was not met is the switching time between white and monochromatic beam. This time is limited primarily by the mechanical drive and can be improved with a more powerful drive motor.

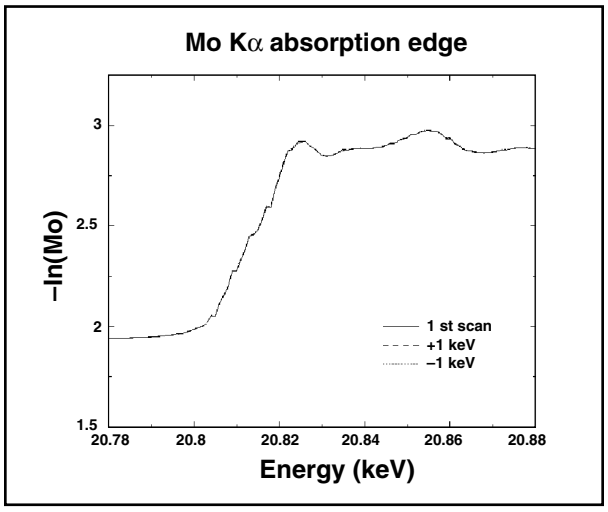

FIG. 2. Three scans of the Mo edge showing no measurable hysteresis after the monochromator was driven ±1 keV from the edge.

Table I. Design goals for the MHATT-CAT microdiffraction monochromator.

Desirable Microbeam Monochromator Property	Goal	Measured
Bandpass for monochromatic	0.02%	0.02%
Bandpass for "white-beam conditions"	>5%	>5%
Energy range	8-22 keV	8-22 keV
Energy accuracy	<2 eV @ 20 keV	<2 eV from 8-21 keV
Mono/white-beam displacement	Negligible	<0.5 μm 14-22 keV
Mono/white-beam parallelism	Negligible	Negligible
Hysteresis during energy scans and when cycling between white and monochromatic conditions	Negligible	Too small to measure
Switching time between mono and white beams	<1 sec	<3 sec

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